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A new technique for temperature measurement and calibration

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Abstract
New working principles generally registered as ISOCALT® improving the current practice of temperature measurement and calibration are presented. The new technique is based on the optimization of the energy circuit associated to the measuring disposition and on the measurement of the temperature gradients around the sensors of the two compared thermometers as basic contributions in the measurement (calibration) uncertainty. These contributions are explained for two commercial types of ISOCALT® devices devoted for calibration of any kind of thermometers on the temperature range of ambient to +500 ^{0}C.
Keywords: template; MSA 2005; papers

1. Introduction

Temperature is one of the basic quantities in all fields of activity. All manufacturing and/or operating processes from laboratory to industrial scale are temperature driven, so stable and accurate measurements are necessary in view to control them in best conditions. The main problem is if the read value on a measuring thermometer having the sensitive element (SE, sensor) immersed in the processing bath is the real value of the bath temperature. This problem is in fact the basic problem of metrology of any measured quantity and may be expressed as finding the uncertainty associated to the read value.
Any kind of processing bath has a distribution in time and space of temperature values, as expressed in terms of temperature gradients. These gradients define the measurement uncertainty associated to a read value and this is the basic idea of the ISOCALT® technique for temperature measurement and calibration [1-3]. Thoroughly data concerning construction and performances of ISOCALT® thermostats are given in technical notes [4] available on web site.
The advantages of the ISOCALT® technique can be well explained by considering the two basic dispositions proposed for accurate calibration.

2. Immersion in an isothermal bath

Dry baths are extensively used in the last years, especially for temperatures over 300 ^{0}C. However, most of the commercial models locate SE of the standard thermometer in the heating mantle and SE of the measuring thermometers in the immersion block, so that important radial temperature gradients exist between them. Additionally, the metallic sheaths of SE create axially temperature gradients. For instance, one of the famous commercial model of temperature calibrator with dry block shows a difference of at least 0.5 ^{0}C at 50 ^{0}C between the built in standard thermometer and another one (Pt-100 with 0.001 ^{0}C resolution) immersed in the block. Such gradients exist also in the fluid baths even in baths for ITS-90 fixed points, so these must be carefully measured in view to estimate the final measurement/calibration uncertainty.
In a recent communication the origins of these gradients are thoroughly reviewed in topoenergetic terms [3] for their better understanding.
Figure 1 shows the axial and cross sections in a cylindrical immersion block of the basic model ISOCALT® 2/70/21, i.e. 2 immersion orifices of 70 mm height. Indices 1 and 2 correspond to the two compared thermometers with the read value T1 and T2, respectively. It results 21 distinct values of temperature differences as combination of 2 measuring points from overall 7 points. These 7 points are located as follows: 3 are placed axially along each immersion orifice (one at the inner bottom and the other ones at L1 and L2 distances; one point is in the centre of the block). On the panel front of the thermostat (see the pictures) these points appear as connecting terminals marked by two figures (the first figure represents the orifice and the second one its position along the orifice). The sign of gradient value shows what the warmer and cooler point is.
By considering that the two SE measuring absolute values of temperature (SEMAV) are perfectly coupled with the immersion block, the main contribution given by temperature gradients to the combined uncertainty of calibration by comparing the TM and TS values in this disposition can be estimated as [3]:

uc = ((Max(Abs(dT1i*(Lo1/Li))))^2 +
(Max (Abs(dT2i*(Lo2/Li))))^2 +
(Max(Abs(dTradial)))^2)^0.5 (1)

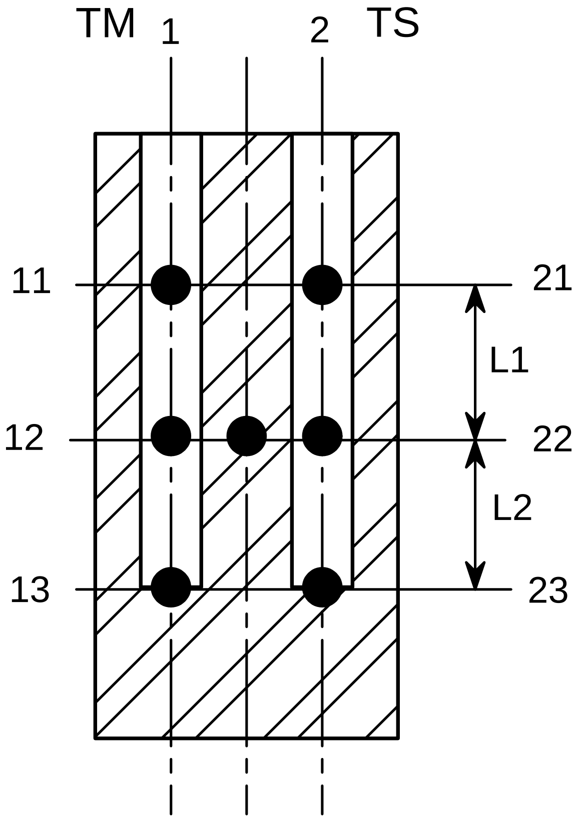

Figure 1.

Calibration in a dry bath.

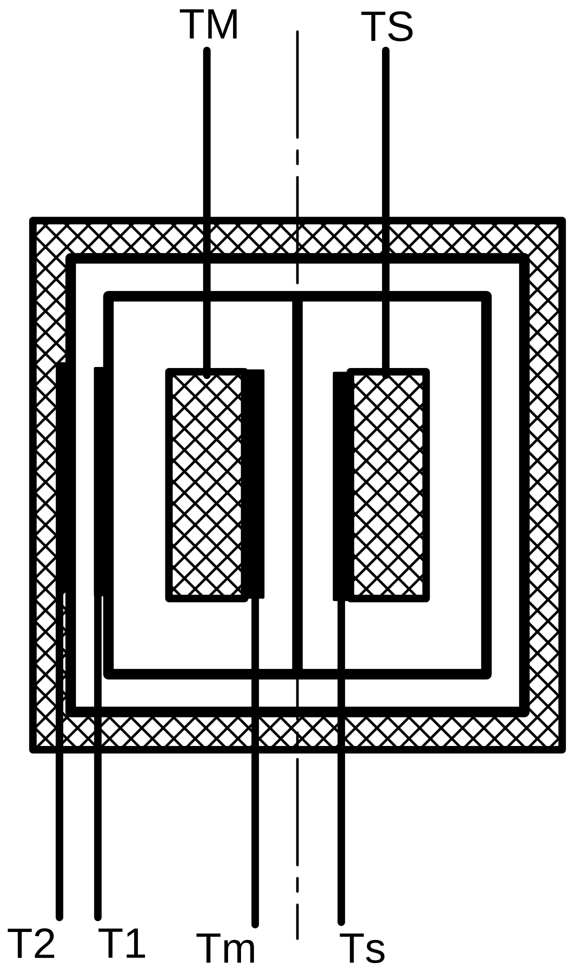

Figure 2.

Calibration in an adiabatic enclosure.

where dT1i/Li and dT2i/Li are the axial gradients along the two orifices, respectively; dTradial are radial temperature differences; Li is the length between each pair of points considered; Lo1 and Lo2 are the length of the two SEMAV, respectively. All dT values are measured by specific SE in differential connections around the two immersion orifices. In fact, uc contains three independent contributions: two of axial and one of radial gradients. Finally, uc will contain other contributions.

The commercial thermostat ISOCALT® 2/70/21 allows the calibration only of one measuring thermometer at once, so metrologists required more “productivity”, namely immersion block with more than 2 orifices. Our prior experience on blocks with many orifices showed unexpected great gradients at equilibrium temperature (dT over 1 ^{0}C), especially at high temperature. However, thoroughly studies allowed finding blocks with 3 and 5 orifices with good results for a wide variety of glass thermometers and pairs of Pt sensors (extensively used in energy meters for warm water).

These thermostats reach the stable temperature in approximately 1 hour on all temperature range (ambient to +500 ^{0}C) with stability of 0.01 ^{0}C (read on standard thermometer) even for months with power consumption at equilibrium ranging between 0 and 100 W depending on the temperature. The electronic block consists in separate modules the same for all thermostats in the series of dry baths and allows measuring digitally dT up to 0.003 ^{0}C. These values are enough taking into account that the usual standard Pt-thermometers have an extended uncertainty of calibration of approximately 0.01 ^{0}C.

Temperature controller has only proportional band digitally controlled by pulse width modulation.

ISOCALT® 2/70/21 was awarded in a series of 20 hi-tec products at the international technical fair TIB-2004 in Bucharest, Romania.

In view to reveal more clear the construction details and the performances of ISOCALT® 2/70/21, Table 1 and 2 present experimental data of comparison of two thermometers having the reading resolution of 0.01 ^{0}C using two identical thermostats which differ by the metal of the immersion block as brass (CuZn37) and Inconel, respectively. The blocks have the outer diameter of 40 mm, 100 mm length, 10 mm the diameter of orifices, and 18 mm the distance between their axes. Iron/constantan thermocouples are used for dT measurements placed in precisely machined holes and insulated with a mixture of oxides. Thermocouples were made, tested and selected by rigorous procedures in view to ensure long life operation and stability on the overall temperature range. For instance, series of 10 independent pairs of thermocouples (wires sampled from the same spools) were selected and tested simultaneously with cold junctions maintained in a water triple point cell, by measuring their emf vs temperature in the range of +20: +600 ^{0}C in 20-30 equidistant points prior and after their annealing at +500 ^{0}C/30 days, so that the same slope was determined as 52.1 ± 0.25 µV/^{0}C. The standard uncertainty of measurement of dT was estimated as being 0.1 µV (under 0.002 ^{0}C).

All investigations with ISOCALT® 2/70/21 using certified standard Pt- thermometer were performed in different laboratories on the dT sensitivity of 0.5 µV/LED corresponding to 0.01 (± 0.005) ^{0}C as the enough highest sensitivity.

Thermometer 1 is a digital thermometer with SEMAV a diode protected by a metallic sheath with 6 mm outer diameter, 5.4 mm inner diameter and 250 mm long. The diode was sealed in epoxy at the end of the rod (Lo1 = 5 mm) and was priory calibrated several times during 2 years in different ISOCALT® thermostats and triple point cells of water by using a certified standard Pt-100 thermometer. Both SEMAV were used without protective metallic sheaths, so that the heating kinetics and temperature stability were thoroughly studied on the all temperature range and especially on ambient to +100 ^{0}C with temperature gradients under 0.01 ^{0}C/20 mm [4].

Thermometer 2 is a glass thermometer with the immersion rod of 8 mm diameter, 120 mm length and the mercury reservoir (SEMAV) of Lo2 = 15 mm long. It results from Table 1 that L1 = 20 mm, L2 = 35 mm and the connecting manner of the 6 temperature points along the orifices, so that temperature differences taken into account dT = (temperature(HI) - temperature(LO)). Only 9 values for dT are considered from the overall of 21 distinct values on radial and axial directions (denoted as Z1, Z2 corresponding to the two orifices, respectively). The read values of the two thermometers (T1 and T2) at different periods of time relative to the start of heating show that the equilibrium is reached in approximately 1 hour for both thermostats and all temperatures. Although both SEMAV are fastened with aluminium foil in orifices, the read values (both absolute and dT values) at equilibrium depend on the connection sheaths of SEMAV to room temperature and on the metal of the immersion block.

Table 2 gives the uc values estimated by considering dT values along SEMAV according to eqn.(1) denoted as SEDT and the overall dT values (OTD) according to the following eqn:

uc = ((Max(Abs(dT(radial))))^2 +
(Max(Abs(dT(Z1))))^2 +
(Max(Abs(dT(Z2))))^2)^0.5 (2).

The resulted uc values are systematically greater for Inconel, for OTD procedure, for thermometer 1 and in all cases they increase with the equilibrium temperature. Good thermal conductive metals for the immersion block, like copper and aluminium alloys, are good enough to obtain good results. These results substantiate the conclusions below. It is important to

Table 1. Experimental values for comparison of two thermometers in two ISOCALT® 2/70/21 thermostats (see the text).

BRASS
11.12.2003

Start: 11:00			Start: 14:00		
Hour	T1	T2	Hour	T1	T2
12:00	60.59	60.58	15:00	90.62	91.40
13:00	60.61	60.58	16:00	90.65	91.40
14:00	60.60	60.58	17:00	90.63	91.40

	HI	LO	L mm	dT ^{0}C	dT/L ^{0}C/mm	dT ^{0}C	dT/L ^{0}C/mm
Radial	12	22	18	0.03	0.00167	0.03	0.00167
	11	21	18	-0.02	-0.00111	-0.05	-0.00278
	13	23	18	-0.03	-0.00167	-0.03	-0.00167
Z1	11	13	55	-0.1	-0.00182	-0.18	-0.00327
	11	12	20	-0.07	-0.0035	-0.08	-0.00229
	12	13	35	-0.03	-0.000857	0.1	0.005
Z2	21	23	55	-0.1	-0.00182	-0.14	-0.00255
	21	22	20	0	0	0.14	0.007
	22	23	35	-0.07	-0.002	-0.14	-0.004

Inconel
09.12.2003

Start: 09:20			Start: 12:30		
Hour	T1	T2	Hour	T1	T2
10:30	59.98	60.50	13:30	88.94	90.20
11:30	60.02	60.50	14:30	88.96	90.20
12:30	60.00	60.50	15:30	88.94	90.20

	HI	LO	L mm	dT ^{0}C	dT/L ^{0}C/mm	dT ^{0}C	dT/L ^{0}C/mm
Radial	12	22	18	0	0	0.01	0.000556
	11	21	18	0.03	0.00167	0.06	0.00333
	13	23	18	-0.02	-0.00111	-0.1	-0.00556
Z1	11	13	55	-0.9	-0.0164	-1.4	-0.0255
	11	12	20	-0.1	-0.005	-0.24	-0.012
	12	13	35	-0.7	-0.02	-1.2	-0.0343
Z2	21	23	55	-0.9	-0.0164	-1.4	-0.0255
	21	22	20	-0.12	-0.006	-0.28	-0.014
	22	23	35	-0.8	-0.0229	-1.2	-0.0343

Table 2. Combined uncertainty (uc) estimated according to OTD and SETD procedures for two thermometers compared in two independent ISOCALT® 2/70/21 having the immersion block made from brass and Inconel. All values are in ^{0}C; values marked as * are obtained in a different day at an interval of approx 1 month (see the text).

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BRASS					Inconel				
T1	T2	T2-T1	OTD	SETD	T1	T2	T2-T1	OTD	SETD
53.72	53.61	-0.11	0.124	0.063	53.91	54.19	0.28	1.205	0.358
60.60*	60.58*	-0.02	0.103	0.044	60.00*	60.50*	0.50	1.273	0.667
65.88	66.06	0.18	0.176	0.093	65.86	66.58	0.42	1.415	0.409
75.64	75.97	0.33	0.205	0.106	75.02	75.91	0.89	1.697	0.452
86.09	86.58	0.49	0.249	0.119	83.68	84.78	1.10	1.982	0.543
90.63*	91.40*	0.77	0.234	0.118	88.94*	90.20*	1.26	1.982	0.552
95.61	96.27	0.66	0.227	0.102	95.94	97.35	1.41	2.409	0.717

note that uc(SETD) takes into account the dT along SEMV, while uc(OTD) considers dT values along the overall orifices, both of them for the same disposition of SEMAV. For further improvements of this new technique, the ratio

FOM = uc(SETD)/uc(OTD) (3)

may define the figure of merit (FOM) of the immersion bath, so that it results from Tables 2 :

FOM (brass) = 0.488 ± 0.038
FOM(Inconel) = 0.284 ± 0.013 (4).

Further analysis of values from these Tables may reveal more important details with deep practical significances.

3. T-calibration in an adiabatic enclosure

One of the basic aims of temperature measurements is as the SEMAV has a uniform temperature. This could be reached by enclosing both SEMAV in equipotential and separate enclosures and considering their temperatures in differential relationship. This difference is in fact the basic contribution in calibration uncertainty in steady state conditions. This disposition is a combination of differential and adiabatic calorimeters [4].
Figure 2 presents the axial cross section of this disposition in which T1 and T2 are the read values for the two thermometers, T1x and T2x are the temperatures of the two equipotential enclosures of SEMAV, Ta and Tb are the temperatures of the outer enclosures maintained as close as possible (Ta-Tb=0). The differential pair connections of (T1x,T2x) and (Ta and Tb) are measured by special and specific SE. It results that the calibration uncertainty estimated by the difference abs(T1x-T2x) is mainly related to:

- the thermal coupling of the two SE to the external medium and
- the controlling accuracy = abs(Ta-Tb).

This disposition has some important advantages, namely: allows small dimensions, small heat capacity and thermal inertia of the overall disposition, easy to realize low temperatures and high accuracy. These make it suitable for fluid baths even for ITS-90 fixed points.
TN 2-2004 describes in more details experimental data obtained for comparison of the same thermometers as above by using the same disposition of high resolution mixing calorimeter [6].
Metallic sheath of thermometer 1 has no sense in this disposition. Even the glass rod of thermometer 2 gives great dT values. Pairs of diodes and diode batteries are compared on the range of ambient to +100 ^{0}C by using thin connecting wires with accuracy of dT down to 10^-5 ^{0}C.

4. Conclusions

The following concluding remarks with practical value can be revealed:

a. The uncertainty associated to a read temperature value is a function of temperature gradients around SEMAV.
b. Measurement of dT can be performed with much higher accuracy and stability, not depending on temperature range, by using simple and cheap SE in differential connection than by absolute temperature.
c. SEMAV of the standard and measuring thermometers must be located in similar conditions in the immersion bath, i.e. in symmetrical locations.
d. In view to minimize the measurement and calibration uncertainties, SEMAV must be thermally coupled as tight as possible with the immersion bath and thermally isolated to the external medium. This means that SE must be immersed without sheath and by using as small (thin) as possible connections.

Further investigations of many users are necessary in view to standardize a commonly adopted procedure, especially for the uncertainties associated to the measurement and calibration using this new technique.

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ISOCALT ® 2/70/21

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MSA has chose Metrology Well Calibrators
from Fluke (Hart Scientific)

“He was incompetent, but because he was caliph people did not observe that”
(Arabian nights)

Metrology Society of Australia (MSA, www.metrology.asn.au) organizes its MSA 2005 convention under slogan: “Smart Measurements: Metrologists Advancing Industry” (Canberra 19-21 October 2005).

GDF-Data Banks was invited (25 July 2005) to present its ISOCALT® products after my first contact to join MSA as member and considering the recent contribution to the 12th International Metrology Congress (20-23 June 2005, Lyon, France). The first version was submitted after 2 days as a short presentation, but the reviewers asked for more and more technical details up to the 5th version on 29 August 2005 when the organizers decided to remove this presentation from the program.

In the same day MSA website advertised the new series of Metrology Well Calibrators from Fluke (Hart Scientific) as the best calibrators on the market (www.hartscientific.com/newstuff/newprod917x.htm).

What are the real reasons of this choice?

> We invite all people honestly interested in correct temperature measurements and calibrations to compare the two series of products. The best way for this purpose is to effectively work with both of them. Did MSA experts work with them? Certainly not, but they already decided! People did not observe that?

We can mention that our first dry block was a copy of Hart Scientific one’s denoted as insert “B” in this new series. In spite of temperature stability this block showed dramatic temperature gradients (1-3 ^{0}C/10 mm) even under 100 ^{0}C. According to our long and intense experience there are necessary several differential sensors around each immersion well in view to establish temperature gradients defining the measuring and calibration uncertainty.

Concerning the uncertainty, this can be established only by users’ experiments, so that for temperature measurements and calibrations it must be related directly to temperature gradients around the sensing elements. The proposed theory for estimation of uncertainties in Fluke Metrology Wells (see their application note) is very far from the real situation of temperature gradients measured by ISOCALT®.

Therefore, read carefully both descriptions first and after that do careful experiments on them. Do not let them to cheat you by tall tales.

Cherchez la femme, monsieurs!

gdf, 05 September 2005

nV-meter

The new type of digital dc nV-meter is specially designed for measuring temperature differences with differential thermocouples. This instrument is low priced, robust, reliable and can optionally replace the similar one µV-meter as module in the electronic block of ISOCALT® series. nV-meter can be used independently for general purposes: calorimetric devices, flowmeters, differential thermometry in medical diagnosis, as high precision null instrument, etc.

nV-meter is an independent instrument (see the attached picture) in a case with the dimensions of W=175 mm, H=70 mm and D=150 mm or in a ISOCALT® standard module with the front panel of W=80 mm, H=130 mm.

nV-meter uses a 3.5 digits panel meter (LED or LCD display) with the full scale of 200 mV (last digit #0.1 mV). The basic amplifier consists in two stages: the first has fixed gain optionally belonging in the range of A1=100-500 and the second one in classical steps of A2=1, 5, 10, 20, 50 and 100.

The actual model has A1=200, so that full scale of the panel meter (±200 mV) corresponds to the input of ±1 mV for A2=1.
In the Table below are given the real units of last digit and full scale in V and ^{0}C for thermocouples Iron-Constantan used in the ISOCALT® series (52.1 ± 0.25 µV/^{0}C, see the previous notes).

A2	Last digit		Full scale	
	nV	^{0}C	µV	^{0}C
1	450	0.01	900	20
2	230	0.005	450	10
5	90	0.002	180	4
10	45	0.001	90	2
20	23	0.0005	45	1
50	9	0.0002	18	0.4
100	4.5	0.0001	9	0.2

nV-meter has a zero adjustment by using a 10-turn precision potentiometer, so that zero is maintained on a long term and on all gain steps. Comparison experiments have been performed on ISOCALT® series by using the modular µV-meter and the new nV-meter and the results were in very good agreement. The main advantages of nV-meter are: (i) it is an independent instrument allowing measurements of temperature gradients in different enclosures simultaneously; (ii) the digital display allows estimating temperature differences and general voltage values with 3 digit accuracy.

Price: 420 USD plus options, taxes and freight.

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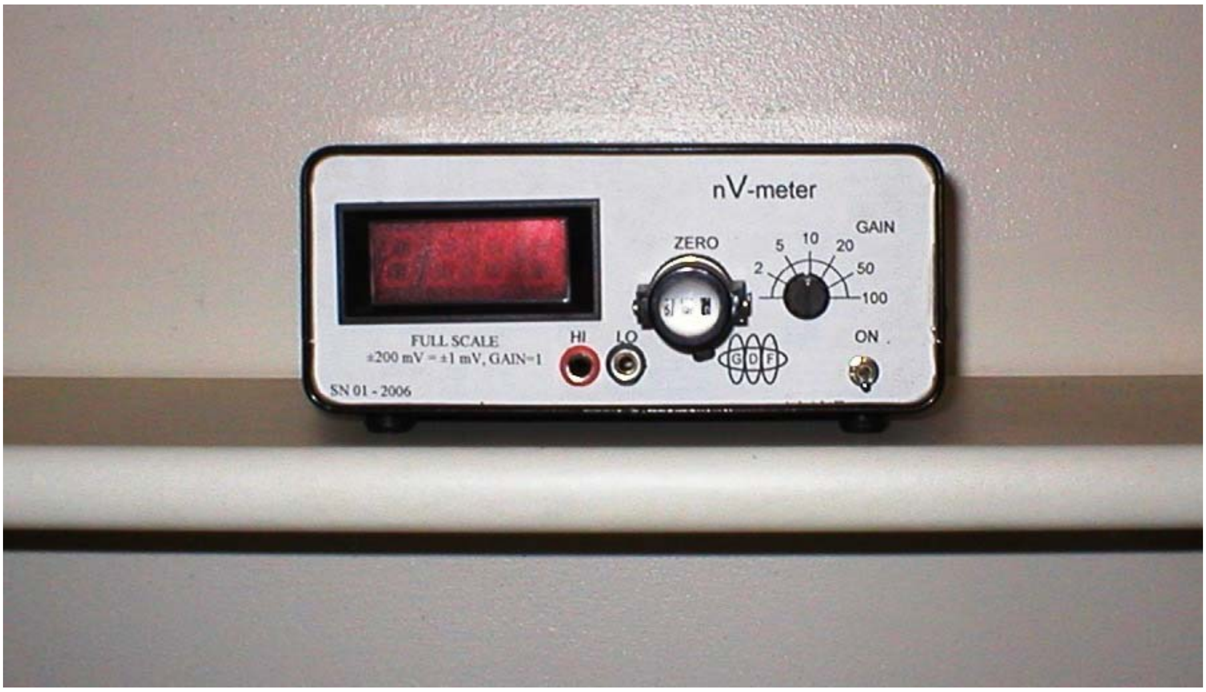

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1997	1	2	Guide of good practice in metrology (Romanian)	AFI
1998	2	1	Editorial: socio-psychological implications in creation and utilization of a databank (Ioan-Bradu Iamandescu); Behavior in vapor-liquid equilibria (VLE): I. Structural aspects; Behavior in vapor-liquid equilibria: II. Several structures in databanks; Symposium on VDC-4 held on 30 October 1997 at Lubrifin-SA, Brasov (Romania).	F
1998	2	2	Practical course of metrology (Romanian)	AFI
1998	2	3	DIFFUTOR-01: Thermally driven diffusion in pure metals	AFI
1998	2	4	VAPORSAT-01: Databanks of thermally driven VLE. The first 100 simple molecules	AFI
1999	3	1	Editorial: New trends in material science: nanostructures (Dan Donescu) DIFFUTOR: Databanks of diffusion kinetics. VAPORSAT: Databanks of vapor-liquid separation kinetics.	F
1999	3	2	Discussions on Applied Metrology	AFI
2000	4	1	Editorial: Laboratory accreditation and inter-laboratory comparisons (Virgil Badescu) Doctoral Theses – important data banks. GDF intends to open new series of experiments on thermo-physical properties. Some comments on uncertainty: global budget and DFT analysis. Events: The 9th International Metrology Congress, Bordeaux, France, 18-21 October 1999.	F
2000	4	2	Measurement and Calibration.	AFI
2001	5	1	Editorial: Metrology ensures moral and technological progress. Topoenergetic aspects of amorphous-crystalline coupling. I. Composite behavior of water and aqueous solutions (paper presented at nanotubes and Nanostructures 2001, LNF, Frascati, Rome Italy, 17-27 October 2001). Events: Nanotubes and nanostructures 2000.School and workshop, 24 September – 4 October 2000, Cagliari, Italy.	F
2001	5	2	Editorial: Viscosity – a symptomatic problem of actual metrology. Visco-Dens Calorimeter: general features on density and viscosity measurements. New vision on the calibration of thermometers: ISOCALT® MOSATOR: Topoenergetic databanks on molten salts properties driven by temperature and composition.	F

continued

Year	VOL	NO	Content (titles)	$*)
2002	6	1	MOSATOR-01: Topoenergetic databanks for one component molten salts; thermally driven viscosity and electrical conductance.	AFI
2002	6	2	Editorial: HuPoTest - Operator calibration or temporal scale psychic test. MOSATOR: topoenergetic databanks of one component molten salts; thermally driven viscosity and electrical conductance.	F
2002	6	3	Editorial: Quo vadis Earth experiment? ISOCALT® : Report on metrological tests	F
2003	7	1	Editorial: Time – an instrument of the selfish thinking. 1st NOTE: Homoeopathy: upon some efficient physical tests revealing structural modifications of water and aqueous solutions. I. Mixing experiments.	F
2004	8	1	Metrological verification and calibration of thermometers using thermostats type ISOCALT® 21/70/2. Metrological verification and calibration of thermometers using thermostats type ISOCALT® 2.2R.	F
2004	8	2	Aspects of correct measurements of temperature. I. measurement of a fixed point according to ITS-90. Physics and Homoeopathy: some physical requirements for homoeopathic practice.(Plenary lecture at the 19th SRH National Congress, 21-22 September 2004, Bucharest, Romania)	F
2005	9	1	AWARD for ISOCALT® at the International Fair TIB-2004, October 2004, Bucharest. ISOCALT® 3/70/21 was awarded in a selection of 20 products by a commission of experts from the Polytechnic University of Bucharest. Upon some aspects of temperature measurements. (12th International Metrology Congress, 20-23 June 2005, Lyon, France)	F
2005	9	2	A new technique for temperature measurement and calibration. National Society of Measurements (NSM). Important warning for T-calibrator users: MSA has chose metrology well calibrators from Fluke (Hart Scientific).	F

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